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IS 2271 (1967): Recommended method for spectrographic analysis of platinum [MTD 10: Precious Metals]



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IS : 2271 - 1967
(Reaffirmed 2001)

Indian Standard

RECOMMENDED METHOD FOR SPECTROGRAPHIC ANALYSIS OF PLATINUM

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Indian Standard

RECOMMENDED METHOD FOR SPECTROGRAPHIC ANALYSIS OF PLATINUM

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The Bombay Bullion Association Ltd, Bombay

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National Test House, Calcutta

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DR T. S. KRISHNAN (Alternate)

SHRI MULCHAND M. SHAH

National Refinery Pvt Ltd, Bombay

DR V. T. SHETH

The Bombay Bullion Association Ltd, Bombay

Indian Standard

RECOMMENDED METHOD FOR SPECTROGRAPHIC ANALYSIS OF PLATINUM

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 18 August 1967, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 In order to determine correctly the various impurities that may be found in platinum used in industry, spectrographic method has been described in this standard.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard recommends spectrographic method for determining impurities in various grades of platinum ranging from 99.00 to 99.99 percent purity.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. SPECTROGRAPHIC METHOD FOR ANALYSIS OF PLATINUM

3.1 Outline of the Method — The platinum metal or the alloy is converted into the ammonium salt and then mixed with a zinc oxide-graphite

*Rules for rounding off numerical values (*revised*).

†Specification for water, distilled quality (*revised*).

buffer in the ratio 2 : 1. Fifteen mg of this mixture are burnt to completion in a dc arc. The resulting spectra give the concentration of the impurity lines in terms of their spectral intensities.

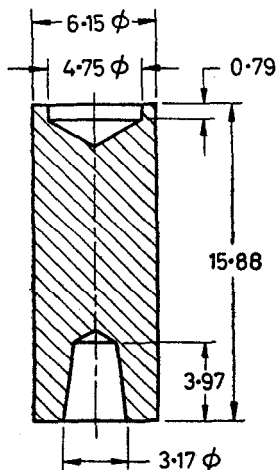
3.2 Apparatus

3.2.1 Spectrograph — Hilger large quartz or equivalent unit.

3.2.2 Condensing Lens — The arc discharge shall be focussed on the collimator lens by means of spherical lens.

3.2.3 Densitometer — Hilger non-recording microphotometer or equivalent.

3.2.4 Electrodes — The upper electrode shall consist of a pointed spectrographically pure graphite rod 3 mm diameter. The lower electrode shall be preformed shallow-cup graphite electrode suitable for 15 mg sample (see Fig. 1).



All dimensions in millimetres.

FIG. 1 PREFORMED GRAPHITE ELECTRODE

3.2.5 Photographic Plate — Ilford ordinary (N/30) or equivalent.

3.2.6 Hilger 7 — step transmission step sector (or equivalent) with known relative sector step openings.

4. PREPARATION OF SAMPLE

4.1 Clean 100 mg of the sample with warm concentrated nitric acid, dissolve in a minimum amount of aqua regia and evaporate to dryness on a

water bath. Dissolve the residue in water. Convert the chloride solution to ammonium chloro-platinate by adding 55 mg of ammonium chloride. Evaporate to dryness. Weigh the residue and mix it with half its weight of a buffer containing equal amounts of zinc oxide and graphite. Use the mixture for taking spectra.

5. STANDARD MIXTURE

5.1 To 2 g of spectrographically pure ammonium chloro-platinate, add 2 mg of each impurity in the form of solution. Dry and mix thoroughly in an agate mortar. This mixture may be taken for all practical purposes as having a concentration of 0.1 percent of each impurity. Mix a buffer containing equal portions of zinc oxide and graphite with twice its weight of the standard and use the mixture for taking spectra. Similarly prepare other standards by altering the quantity of impurities to be added as to conform to the required concentration ranges.

5.2 A series of nine standards with impurities as shown in Table 1, may be prepared to cover the range from 0.001 to 0.1 percent. Other standards of different concentrations and impurity elements may be prepared, if found necessary for any special type of work.

TABLE 1 STANDARD SAMPLES

STANDARD SAMPLE	ELEMENT, PERCENT									
	Pb	Sn	Fe	Ni	Pd	Au	Ir	Cu	Ag	Rh
1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
2	0.075	0.075	0.075	0.075	0.075	0.075	0.075	0.075	0.075	0.075
3	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
4	0.025	0.025	0.025	0.025	0.025	0.025	0.025	0.025	0.025	0.025
5	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
6	0.0075	0.0075	0.0075	0.0075	0.0075	0.0075	0.0075	0.0075	0.0075	0.0075
7	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005
8	0.0025	0.0025	0.0025	0.0025	0.0025	0.0025	0.0025	0.0025	0.0025	0.0025
9	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001

6. PROCEDURE

6.1 Load the lower electrode (*see* 3.2.4) with 15 mg of sample (*see* 4.1) or standard (*see* 5.2) for taking spectra under conditions given in Table 2. Duplicate or triplicate exposures shall be made of each standard on each plate and triplicate exposures of the sample under test. Fresh samples and electrodes shall be used for each exposure. Each plate shall also include a step sector spectrum of the iron arc, run at 3 A with an exposure of 30 seconds.

6.2 Using the non-recording microphotometer, measure the densities of the line as well as background on either side, for each analytical and internal standard line given in Table 3. Similarly measure the peak densities for each step of a suitable iron line (2 679·07 Å) in the step sector spectrum for plate calibration.

6.3 Plot the density of the step sector iron line against the log of the relative sector step openings. This gives the plate calibration curve.

6.4 Using the plate calibration curve (*see* 6.3) convert all the densities (*see* 6.2) to relative intensities. Make the background correction by subtracting the average background intensity from the line intensity. Plot the intensity ratio of the analytical line to the appropriate internal standard line against the percent concentration on a log-log scale to give the analytical working curve for each impurity element. Obtain concentrations of impurities in samples from the corresponding intensity ratio.

TABLE 2 SPECTROGRAPHIC CONDITIONS

(Clause 6.1)

CONDITIONS	UNITS
(1)	(2)
Slit length	2·0 mm
Slit width	0·015 mm
Wavelength range	2 400 Å - 3 450 Å
Current	10 A dc
Arc gap	4·0 mm
Pre-arcing period	Nil
Exposure	20 seconds
Processing	Develop for 180 seconds at 20°C in Metol-Hydroquinone developer, rinse in water, and fix for 10 minutes. Wash the plate for 15 minutes in running water and finally rinse it in distilled water before drying.

NOTE — For calcium line, spectral region shall be adjusted.

TABLE 3 LIST OF SUITABLE LINES APPLICABLE TO LARGE QUARTZ SPECTROGRAPH

(Clause 6.2)

ELEMENT	INTERNAL STANDARD LINE	ANALYTICAL LINE (OVER 99.5% PLATINUM)	ANALYTICAL LINE (99.0-99.5% PLATINUM)
(1)	(2)	(3)	(4)
Aluminium	Pt 2 574.49	3 082.16	2 575.1
Antimony	Pt 2 574.49	2 877.92	2 877.92
Bismuth	Zn 2 670.53	3 067.72	2 938.30
Calcium	Pt 2 574.49	4 226.73	3 006.86
Chromium	Pt 2 574.49	2 843.25	2 843.25
Cobalt	Pt 2 574.49	2 521.36	2 521.36
Copper	Zn 2 670.53	3 247.53	2 492.3
Gold	Zn 2 670.53	2 675.95	2 675.95
Indium	Zn 2 670.53	3 039.36	3 039.36
Iridium	Pt 2 574.49	2 543.97	2 543.97
Iron	Pt 2 574.49	2 720.91	2 720.91
Lead	Zn 2 670.53	2 833.06	2 833.06
Magnesium	Zn 2 670.53	2 776.69	2 776.69
Manganese	Pt 2 574.49	2 605.68	2 605.68
Molybdenum	Pt 2 574.49	2 816.15	2 816.15
Nickel	Pt 2 574.49	3 012.00	3 012.00
Palladium	Pt 2 574.49	3 242.7	3 027.9
Rhodium	Pt 2 574.49	3 434.89	3 434.89
Silicon	Pt 2 574.49	2 506.9	2 435.16
Silver	Zn 2 670.53	3 382.89	—
Tin	Zn 2 670.53	2 839.98	2 839.98
Tungsten	Pt 2 812.98	2 724.35	2 724.35
Vanadium	Zn 2 670.53	3 183.98	3 183.98

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INDIAN STANDARDS INSTITUTION

Headquarters

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 1

Telephones 27 36 11 - 20

Telegrams Manaksanstha

Branch Offices

Telegrams Manaksanstha

534 Sardar Vallabhbhai Patel Road
 5 Chowringhee Approach
 117/418 B Sarvodaya Nagar
 54 General Patters Road

Bombay 7
 Calcutta 13
 Kanpur
 Madras 2

Telephone 35 70 27
 " 23-18 23
 " 3 76 95
 " 8 72 78

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